

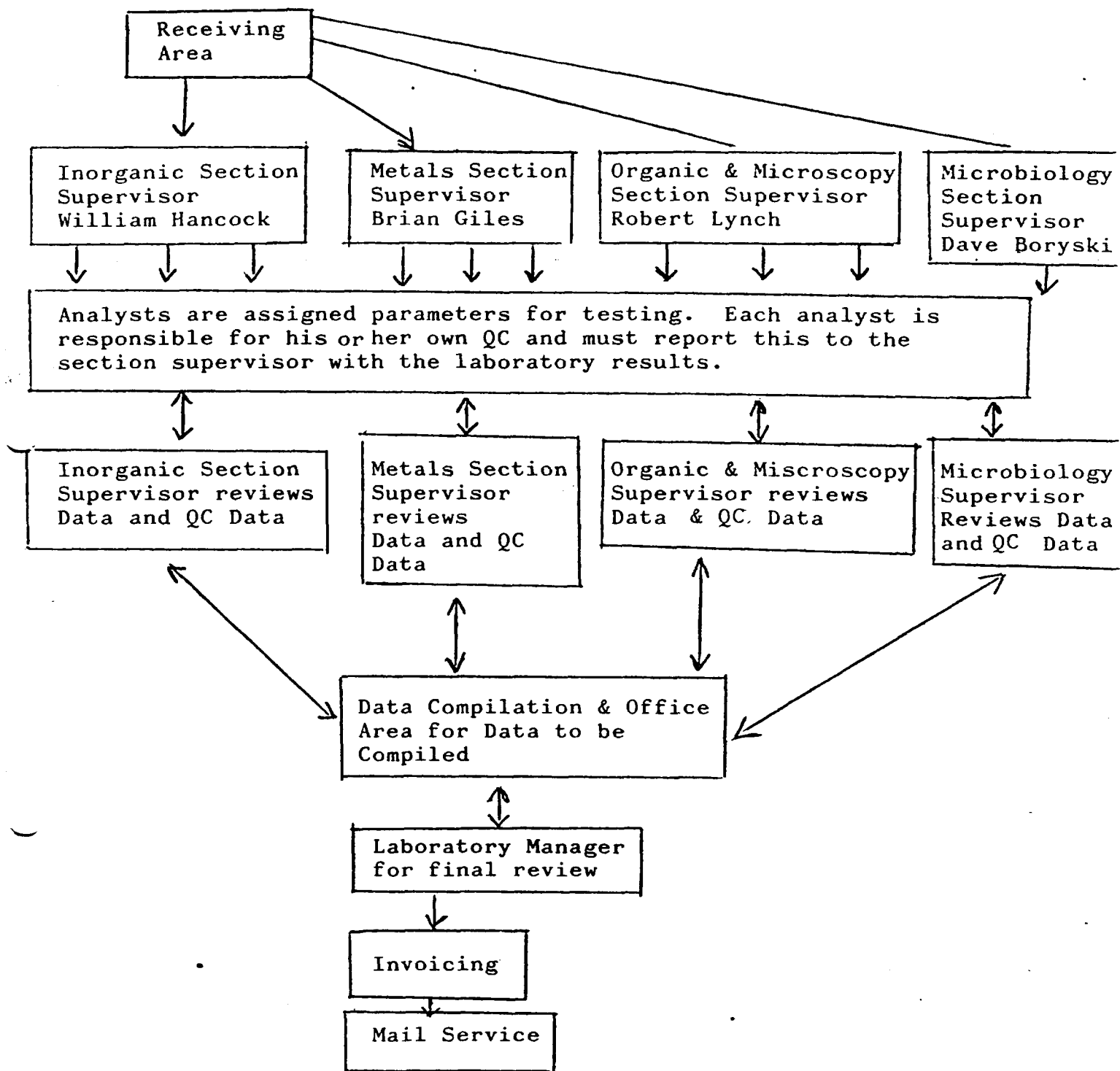
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SUPPLEMENT TO
QUALITY ASSURANCE
MANUAL
OCTOBER 4, 1984

CAL

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SECTION 5.4



NOTE: That in the last phases of data review there is a continuous interaction between the laboratory manager and the section supervisors. This is necessary to review any outlier QC data and to decide the corrective to be taken.

SECTION 5.5

The following are current precision and accuracy charts for water and soil samples used by our laboratory for the listed parameters and ranges.

Parameter	Precision		Accuracy		Completeness	
	Water %	Sediment%	Water %	Sediment %	Water %	Sediment %
Asbestos % of Bulk		±20				
Asbestos filter/liters	±10					
Aluminum	±20	±25	± 25	±35	99	88
Chromium	±15	±20	± 20	±30	96	90
Barium	±20	±30	± 25	±35	96	85
Beryllium	±10	±15	± 15	±19	95	92
Cobalt	±20	±30	± 20	±25	94	80
Copper	±10	±20	± 15	±20	94	88
Iron	±12	±25	± 20	±30	97	91
Nickel	±10	±15-	± 20	±25	92	80
Manganese	±10	±20	± 15	±25	95	90
Zinc	±20	±27	± 18	±23	96	84
Vanadium	±22	±30	± 20	±26	92	84
Silver	±10	±30	± 25	±40	70	50
Arsenic	±25	±25	± 30	±30	85	85
Antimony	±15	±20	± 20	±23	95	90
Selenium	±20	±30	± 25	±32	94	86
Thallium	±18	±25	± 14	±18	102	96
Mercury	±20	±35	± 25	±35	94	85
Tin	±25	±35	± 26	±30	101	82

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SECTION 5.5 cont.

Parameter	Precision		Accuracy		Completeness	
	Water %	Sediment %	Water %	Sediment %	Water %	Sediment
Cadmium	± 20	± 22	± 15	± 18	99	96
Lead	± 20	± 20	± 11	± 20	95	80
Boron	± 20	± 25	± 17	± 25	90	75
Ammonia-Nitrogen	± 20	± 30	± 20	± 27	98	87
Cyanide	± 10	± 25	± 20	± 30	100	80
Sulfides as S	± 20	± 35	± 25	± 38	91	82
Purgables	± 15	± 15	± 20	± 20	95	90
Base Neutrals	± 20	± 25	± 25	± 30	90	80
Acid Extracts	± 15	± 25	± 20	± 30	90	80
Pesticies	± 15	± 20	± 15	± 20	90	80
PCB's	± 10	± 20	± 15	± 20	95	85
TOC	± 5	± 20	± 5	± 20	97	90

SECTION 5.7

The preservatives used in the field are placed in the appropriate containers in the laboratory before shipment of the sample containers to the customers. These preservatives are all reagent grade chemicals stored in original containers. The nitric acid used for metals is Baker brand reagent grade for use in trace metal analysis. If the preservative is to be mixed with equal parts of water this is done immediately preceeding the introduction of the mixed preservative into the sample containers. Any left over mixture of acid and water is disposed of in the proper manner.

SECTION 5.8

Calibration standards are made from stock standards. On a regular basis the stock standards are either made from high purity material or purchased from a supplier. All stock standards purchased are certified by the manufacturer. The specific methods and frequencies for making standards for the different tests are detailed in the following paragraphs. All stock standards are dated when received or made. All calibration standards are dated when made. If any drift or response from a calibration standard is detected, a new standard is made up and checked against the old standard. If the old standard is found to be sub-standard it is discarded. If the drift or response change is not due to the standard, corrective action to determine the problem must be taken.

Most metal stock standards are made from reagent grade compounds or pure metals. The balance of the stock standards are purchased from a manufacturer and are certified. They are dated and kept for one year. Purchased standards are replaced during the month of their expiration date. Calibration standards are made monthly and dated. Calibration standards for most metals range from 0.5 mg/l to 10 mg/l. The alkali metals are calibrated between 0 and 50 mg/l. Mercury, Arsenic, and Selenium are calibrated between 0 and 10 ug/l. Antimony is calibrated between 0 and 30 ug/l. All low level standards for furnace work are made up fresh and disposed of after use.

All stock standards for conventional parameters are made from reagent grade compounds. The shelf life of these standards vary from compound to compound. All working standards are made from the stock standards as necessary. Each working standard is initialed and dated by the person who makes it. Every time a working standard is made it is standardized by the method determined for that compound. If a working standard is found to be changing (from a drift in the method standard recovery) a new working standard is made. If the new working standard does not perform as is required, if the shelf life of this new standard is seemingly short, or if any aspects or outcomes of the tests become questionable a new stock standard is made immediately. Calibration or working standards are made to encompass all sample concentrations. If a sample is outside of the working range it is diluted and the test is repeated on the dilution. All calibration curves use at least three calibration points.

Volatiles

Pure standards of the individual compounds with purity > 96% are used for all standards. Standards are prepared monthly in methanol and placed into 5ml vials with teflon lined caps and stored refrigerated.

Soil - standards are made to give concentrations of 1 to 3 mg/kg for aromatic and 2 to 5 for chlorinated compounds.

Water - standards are made to give concentrations of 10 to 20 ug/l for aromatic and 20 to 40 for chlorinated compounds.

Spikes are run on every 10th or less set of samples for both water and soil and contain the following:

Chlorinated: Chloroethane
Methylene chloride
Chloroform
1,1,1-Trichloroethane
Trichloroethene
Tetrachloroethene
Chlorobenzene
1,2- Dichlorobenzene

Aromatic:	Benzene	Ethylbenzene
	Toluene	1,3,-Dichlorobenzene
	Chlorobenzene	

The spikes are made from the same materials as the standards. Spike levels are in the same ranges as the standards.

Detection limits	Water	Soil
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SAMPLE ID
PARAMETER- 601VOAs
MATRIX- WATER
INJECTION VOLUME 10.000ml

	mg/l	mg/l
Chloroform		(0 .004
Bromodichloromethane		(0 .008
Dibromochloromethane		(0 .008
Bromoform		(0 .01
Bromomethane		(0 .008
Dichlorodifluoroethane		(0 .01
Chloroethane		(0 .002
Methylenechloride		(0 .004
Trichlorofluoromethane		(0 .008
1,1-Dichloroethene		(0 .002
Chloromethane		(0 .005
1,1-Dichloroethane		(0 .002
trans-1,2-Dichloroethene		(0 .002
1,2-Dichloroethane		(0 .002
1,1,1-Trichloroethane		(0 .002
Carbontetrachloride		(0 .008
1,2-Dichloropropane		(0 .002
trans-1,3-Dichloropropene		(0 .002
Trichloroethene		(0 .002
cis-1,3-Dichloropropene		(0 .002
1,1,2-Trichloroethane		(0 .002
2-Chloroethylvinylether		(0 .005
1,1,2,2-Tetrachloroethane		(0 .002
Tetrachloroethene		(0 .002
Vinyl chloride		(0 .005


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*****
SAMPLE ID                               DATE
PARAMETER- 602VOAs
MATRIX- WATER
INJECTION VOLUME  10.000ml
*****

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	mg/l	mg/l
Benzene		(0 .0002
Toluene		(0 .0002
Chlorobenzene		(0 .0002
Ethylbenzene		(0 .0002
Xylene		(0 .0002
1,2-Dichlorobenzene		(0 .0004
1,3-Dichlorobenzene		(0 .0004
1,4-Dichlorobenzene		(0 .0004


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*****
SAMPLE ID                               DATE
PARAMETER- 602VOAs
MATRIX- SOIL
EXTR. DATE                             METHOD Head space
SAMPLE SIZE   3.2000gm                 EXTR. VOL   0.00ml
DILUTION =1:      1                     FINAL VOLUME 0.00ml
INJECTION VOLUME 0.500ml
*****

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	mg/kg	mg/kg
Benzene		< .02
Toluene		< .02
Chlorobenzene		< .02
Ethylbenzene		< .02
Xylene		< .02
1,2-Dichlorobenzene		< .04
1,3-Dichlorobenzene		< .04
1,4-Dichlorobenzene		< .04

Base/Neutral Fraction:

For individual parameter groups compounds of > 95% purity are used for preparing standards in the appropriate solvent. For screening purposes, prepared standard mixtures are purchased. New concentrated standards are prepared every six months and spikes and standards stored refrigerated in amber bottles with teflon lined caps.

Spikes and duplicates are run on a minimum 10% of the samples. Spike levels for the compounds used are at 0.1mg/l for water and 5mg/kg in soil. The compounds spiked are:

- 1,4-Dichloroethane
- Bis (2-Chloroisopropyl) ether
- Nitrobenzene
- Napthalene
- Dimethylphthalate
- 2,4-Dinitrotoluene
- Phenanthrene
- Dibutylphthalate
- Pyrene
- Bis (2-ethyl hexyl) phthalate

	<u>Detection Limits</u>	
	<u>Water(mg/l)</u>	<u>Soil (mg/l)</u>
Acenaphthene	<0.005	<0.1
Acenaphthylene	<0.005	<0.1
Anthracene	<0.005	<0.1
Benzo(a)anthracene	<0.005	<0.1
Benzo(b)fluoranthene	<0.005	<0.1
Benzo(k)fluoranthene	<0.005	<0.1
Benzo(a)pyrene	<0.005	<0.1
Benzo(ghi)perylene	<0.005	<0.1
Benzyl butyl phthalate	<0.005	<0.1
Bis(2-chloroethyl)ether	<0.005	<0.1
Bis(2-chloroethoxy)methane	<0.005	<0.1
Bis(2-ethylhexyl)phthalate	<0.005	<0.1
Bis(2-chloroisopropyl)ether	<0.005	<0.1
4-Bromophenyl phenylether	<0.005	<0.1
2-Chloronaphthalene	<0.005	<0.1
4-Chlorophenyl phenylether	<0.005	<0.1
Chrysene	<0.005	<0.1
Dibenzo(a,h)anthracene	<0.005	<0.1
Di-n-butylphthalate	<0.005	<0.1
1,3-Dichlorobenzene	<0.005	<0.1
1,2-Dichlorobenzene	<0.005	<0.1
1,4-Dichlorobenzene	<0.005	<0.1
3,3'-Dichlorobenzidine	<0.01	<1
Diethyl phthalate	<0.005	<0.1
Dimethyl phthalate	<0.005	<0.1
2,4-Dinitrotoluene	<0.005	<0.1
2,6-Dinitrotoluene	<0.005	<0.1
Di-n-octylphthalate	<0.005	<0.1
Fluoranthene	<0.005	<0.1
Fluorene	<0.005	<0.1
Hexachlorobenzene	<0.005	<0.1
Hexachlorobutadiene	<0.005	<0.1

	<u>Detection Limits</u>	
	<u>Water(mg/l)</u>	<u>Soil (mg/l)</u>
Hexachlorethane	<0.005	<0.1
Indeno(1,2,3-cd)pyrene	<0.005	<0.1
Isophorone	<0.005	<0.1
Naphthalene	<0.005	<0.1
Nitrobenzene	<0.005	<0.1
N-Nitrosodi-n-propylamine	<0.005	<1
Phenanthrene	<0.005	<0.1
Pyrene	<0.005	<0.1
1,2,4-Trichlorobenzene	<0.005	<0.1
Benzidine	<0.1	<1
Hexachlorocyclopentadiene	<0.010	<1
N-Nitrosodimethylamine	<0.005	<1
N-Nitrosodiphenylamine	<0.005	<1

Phenols

Pure standards of individual compounds with purity of >96% are used for standards. Concentrated standards are prepared every six months in 2-propanol with dilutions for working standards and spikes prepared monthly, and stored refrigerated in amber bottles with teflon lined caps. Spikes and duplicates are run on every 10th or less set of samples for both water and soil. Both spikes contain the following:

	Spike Levels	
	Water (ug/l)	Soil (mg/kg)
2-Chlorophenol	47	2.3
2-Nitrophenol	82	4.1
Phenol	340	17
2,4-Dimethylphenol	360	18
2,4-Dichlorophenol	74	3.7
2,4,6-Trichlorophenol	113	56
4-Chloro-3-methylphenol	180	90
Pentachlorophenol	1060	53
4-Nitrophenol	930	46

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SAMPLE ID                               DATE
PARAMETER- PHENOLS
MATRIX- WATER
EXTR. DATE                             METHOD Liquid/liquid
SAMPLE SIZE    0.5000L                 EXTR. VOL    1.00ml
DILUTION =1:    1                     FINAL VOLUME 1.00ml
INJECTION VOLUME 0.004ml
*****

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	ug/ml	mg/l
2-Chlorophenol		(.0005
2-Nitrophenol		(.0005
Phenol		(.0002
2,4-Dimethylphenol		(.0004
2,4-Dichlorophenol		(.0003
2,4,6-Trichlorophenol		(.0006
2,4,5-Trichlorophenol		(.0006
4-Chloro-3-methylphenol		(.0004
2,3,5-Trichlorophenol		(.0006
2,4-Dinitrophenol		(.015
2-Methyl-4,6-dinitrophenol		(.02
Pentachlorophenol		(.007
4-Nitrophenol		(.003

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*****
SAMPLE ID                               DATE
PARAMETER- PHENOLS
MATRIX- SOIL
EXTR. DATE                             METHOD Soxhlet
SAMPLE SIZE  10.0000gm                 EXTR. VOL    1.00ml
DILUTION =1:    1                     FINAL VOLUME 1.00ml
INJECTION VOLUME 0.004ml
*****

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	ug/ml	mg/kg
2-Chlorophenol		(.02
2-Nitrophenol		(.02
Phenol		(.01
2,4-Dimethylphenol		(.01
2,4-Dichlorophenol		(.01
2,4,6-Trichlorophenol		(.03
2,4,5-Trichlorophenol		(.03
4-Chloro-3-methylphenol		(.02
2,3,5-Trichlorophenol		(.03
2,4-Dinitrophenol		(.02
2-Methyl-4,6-dinitrophenol		(.6
Pentachlorophenol		(.3
4-Nitrophenol		(.2

Pesticides

Pure compounds of >96% purity are used for all standards. Concentrated standards are prepared every six months in iso-octane. Dilutions for working standards and spike from these. All are stored refrigerated in amber bottles with teflon lined caps.

Spikes and duplicates are run every 10% or less, for each set of samples, for each matrix. Spikes contain the following:

	Spike Conc.	
	Water (<u>ug/l</u>)	Soil (<u>mg/kg</u>)
Lindane	1	0.05
Aldrin	0.5	0.05
Endrin	0.5	0.05
p,p'-DDE	0.5	0.05
p,p'-DDT	1	0.05
Endosulfon I	1	0.05

PCBs

Standards are obtained of >96% purity for individual Arochlors. Concentrated standards are prepared every six months in iso-octane and stored refrigerated. Dilutions are made from these standards for

1. Calibration standards - at 0.1, 0.4, 1.0ng/ml
2. Water spike = to give 10 ug/l
3. Soil, sediment spike - to give $\frac{1\text{mg}}{\text{kg}}$

Spikes and duplicates are run on every 10th or less sets of samples for both soil and water.

SAMPLE ID ANAL. DATE:
FOR PCB IN WATER BY Liquid/liquid EXTR DATE
SAMPLE SIZE: 0.50L EXTRACT VOL: 5.0ml
DILUTION=1: 1 FINAL VOL= 5ml
INJECTION VOLUME .004 ml

Total ng injected = 0.00
PCB Concentration = (.0004 mg/l

* *****

SAMPLE ID ANAL. DATE:
FOR PCB IN SOIL BY Soxhlet EXTR DATE
SAMPLE SIZE: 10.00gm EXTRACT VOL: 5.0ml
DILUTION=1: 1 FINAL VOL= 5ml
INJECTION VOLUME .004 ml

Total ng injected = 0.00
PCB Concentration = (.04 mg/kg

SECTION 5.9

The CAL manual lists routine detection limits and the methods employed to reach these limits. To satisfy your requirements for detection limits for Chromium, Cadmium, and Lead we will use a graphite furnace technique instead of direction aspiration. The methods and detection limits are as follows:

Parameter	Method	Detection Limit (ug/l)
Cadmium	213.2	1.0
Chromium	218.2	2.0
Lead	239.2	5.0

These methods are all found in Methods for the Chemical Analysis of Water and Wastes EPA 600/4-79-020, U.S. Environmental Protection Agency March 1979. WE do not keep precision and accuracy charts for furnace analysis.

SECTION 5.14 and 5.15

The methods for finding and treating outliers is found in section 7 of the Canton Analytical Laboratory Quality Assurance Manual. All quality control data is reported to a single person in charge of entering this data into precision and accuracy charts. (Shown on page 24 of the Quality Assurance Manual. These precision and accuracy charts are continous records of performance for individual parameters and their respective ranges. These records are kept seperately for water, sludge, EP Toxicity, sediment, miscellaneous liquids, and miscellaneous solids. From these records critical differences between duplicates and upper and lower control limits for spike recovery are calculated, tabulated and distributed to the key people involved in data assessment. These tables are used to find and assess both precision and accuracy outliers. The method standards and blanks are recorded as an effort to spot trends in calibration or instrument drift, or in reagent shelf life. These tables can also be used to help the analyst see their ability to master and perform standard methods. All outliers are brought to the attention of the laboratory manager and section managers. The data is then assessed and corrective actions are taken. Once the problem has been located the proper corrections are made to rectify the data acceptability.